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# **NORTHERN**

Utilization Research & Development Division

## **Publications and Patents**

**July - December 1969**

PROCUREMENT SECTION  
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Northern Utilization Research and Development Division  
Agricultural Research Service  
United States Department of Agriculture  
1815 North University  
Peoria, Ill. 61604





## INTRODUCTION

The Congress in 1938 authorized four regional laboratories, now known as Utilization Research and Development Divisions, to conduct basic and applied research designed to expand, improve, and develop through science and technology the utilization of American farm crops. The need and importance of such research arise because the farmer is not organized to carry on modern scientific research to maintain old markets for his products and to create new ones. Since their inauguration, these laboratories have contributed much basic knowledge of the chemical composition and physical properties of farm commodities and have applied this knowledge to create new or improved products and processing technology that have enhanced utilization of many farm commodities.

In 1969, the Southeastern Agricultural Research Laboratory, headquarters of the Southeastern Utilization Research and Development Division, was built to serve the specialized needs of that region.

The Northern Utilization Research and Development Division is responsible for research on industrial utilization of the cereal grains — corn, wheat,

barley, grain sorghum, and oats; and the oilseeds — soybeans and flaxseed. Except for wheat and barley, the research includes food and feed uses of these crops. In the Department's program of research on replacement crops, the Northern Division conducts all screening and characterization studies on uncultivated plants and their components. It is also responsible for more intensive research on new oilseeds containing erucic acid and on new gum and pulp fiber plants. In addition to its internal program of research, it carries out work through domestic contracts and grants and conducts related research abroad under grants or contracts involving Public Law 480 funds.

The research investigations at the Northern Division are supported by more than 450 people, about one-half of whom have professional status. This body of highly trained men and women with specialized knowledge in various disciplines are responsible for the scientific publications and patents listed here.

General information about these four Divisions is given inside the back cover.

## REQUEST FOR INFORMATION

The results of research of the Northern Utilization Research and Development Division are published regularly in the technical literature, and public-service patents are secured to cover patentable inventions and discoveries (see page 45). As a convenient guide to our publications and patents, a list with abstract is published semi-annually. The abstracts describe the current research and indicate the progress achieved. Further information on any of the developments, as well as earlier technical papers, may be obtained by writing us.

In conformance with the policy of the Department of Agriculture, Northern Division publications are available to scientists and other specialists, librarians, representatives of the press, and others interested.

Requests for specific reprints should be by number and addressed to the Northern Division. Those titles marked with an asterisk [\*] are not available for distribution.

Most of the publications are in journals that are available in libraries. Photographic copies of most journal articles on research at this Division can be purchased from the National Agricultural Library of the U.S. Department of Agriculture, Washington, D.C. 20250.

No publications will be sent regularly in response to foreign requests unless exchange arrangements have been made with the Director of the National Agricultural Library.

Copies of previous lists of publications and patents are available upon request.



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## PUBLICATIONS

[Publications marked with an asterisk (\*) are not available for distribution. When requesting reprints, please order by number.]

- 2530** • **Unsymmetrical Dithiobis(thioformates)**  
B. S. SHASHA, W. M. DOANE, C. R. RUSSELL, and C. E. RIST  
Carbohydr. Res. 10(3): 449-445. July 1969

Previously unknown, unsymmetrical dithiobis(thioformate) sugar derivatives were prepared by two routes and characterized; also some of their properties were investigated.

- 2531** • **Relative Reduction Rates of Fatty Acid Isomers by Hydrazine**  
C. R. SCHOLFIELD, R. O. BUTTERFIELD, T. L. MOUNTS, and H. J. DUTTON  
J. Amer. Oil Chem. Soc. 46(6): 323-326. June 1969

Mixtures of fatty acids and of hydrocarbons have been reduced by hydrazine in ethyl alcohol solution at 50° C. Samples were removed during reduction for analysis and the relative reduction rates calculated by digital computer. *cis*-15-Octadecenoic acid is reduced 1.3 times as fast as its *cis*-9-

isomer. No differences were found among *cis*-6-, -9- and -12-octadecenoic acids. *cis*-3-Octadecene was also reduced 1.3 times as fast as the *cis*-9-isomer and *cis*-1-octadecene 8.6 times as fast. Elaidic acid was reduced 1.27 times as fast as oleic.

- 2532** • **Preparation and Plasticizing Characteristics of Some *N, N*-Disubstituted Amides of Erucic and Crambe Acids**  
R. R. MOD,<sup>1</sup> F. C. MAGNE,<sup>1</sup> EVALD L. SKAU,<sup>1</sup> H. J. NIESCHLAG, W. H. TALLENT, and I. A. WOLFF  
(<sup>1</sup>South. Util. Res. Develop. Div., New Orleans, La.)  
Ind. Eng. Chem., Prod. Res. Develop, 8(2): 176-182. June 1969

Selected long-chain fatty amides of erucic and rapeseed oil fatty acids have proved to be good plasticizers for vinyl chloride resins. Erucic acid itself or its natural source, rapeseed oil, is currently imported. Now that erucic acid can be obtained domestically and more abundantly from crambe oil, plasticizer evaluations were extended to include

mixed crambe acid derivatives and a wider spectrum of amine moieties. Amides were also prepared and evaluated from crambe oil that had been selectively hydrogenated to remove polyunsaturation. Direct aminolysis was investigated as a potentially more economical route to amide plasticizers from seed oils.

2533 • **L-Asparaginase Production by Various Bacteria**

R. E. PETERSON and A. CIEGLER

Appl. Microbiol. 17(6): 929-930. June 1969

L-Asparaginase inhibits tumor cell growth. Clinical trials have been dependent on production of this enzyme from the bacteria *Escherichia coli*. Reported yields from this organism have been as high as 950 IU/gram (dry weight of cells). A screen of various bacteria was initiated to find other organisms that could provide high yields of L-asparaginase. One hundred twenty-three bacteria were

screened for L-asparaginase production. Of these, *Erwinia aroideae* NRRL B-138 was selected for further studies and found capable of producing yields of 1,250 IU/gram (dry weight of cells). This L-asparaginase was found to be immunologically distinct from the enzymes produced by *Escherichia coli* and *Serratia marcescens*.

2534 • **13-Aminotridecanoic Acid from Erucic Acid**

JOSEPH L. GREENE, JR.,<sup>1</sup> ROBERT E. BURKS, JR.,<sup>1</sup> and

IVAN A. WOLFF

(<sup>1</sup>Southern Research Institute, Birmingham, Ala.)

Ind. Eng. Chem., Prod. Res. Develop. 8(2): 171-176. June 1969

13-Aminotridecanoic acid, the monomer for nylon 13, was synthesized from commercial erucic acid or its derivatives by several methods in a study to determine the method of choice, which involved oxidative ozonolysis of erucitrile, followed by esterification before isolation to give methyl 12-cyanodecanoate. The cyano ester was then catalytically reduced and hydrolyzed, without isolation of

intermediates, to give very pure 13-aminotridecanoic acid in 55 to 60% yield from the erucitrile. Almost equally convenient was a method starting with oxidative ozonolysis of methyl erucate to the half ester of brassylic acid, which was then converted successively to the amide-ester, nitrile-ester, amino ester, and amino acid. Other methods tried were significantly less desirable because of low yields and difficult purifications.

2535 • **Antibacterial Compound from a Soybean Product Fermented by *Rhizopus oligosporus***

HWA L. WANG, DORIS I. RUTTLE, and C. W. HESSELTINE

Proc. Soc. Exp. Biol. Med. 131(2): 579-583. June 1969

Strains of *Rhizopus oligosporus* produce an antibacterial compound especially active against some gram-positive microorganisms. The material can be extracted with water from soybeans fermented by *R. oligosporus*. It also can be recovered from culture broth by ammonium sulfate precipitation. The compound is fairly stable in its semipurified state

and may consist of four or five components.

The antibacterial activity produced by a mold commonly used in an Oriental food fermentation is considered significant for those people whose diets are often nutritionally inadequate. Conceivably, ingestion of this antibacterial material may confer disease resistance.



- 2536 • **A Candidiasis in Larvae of the Douglas-fir Tussock Moth,**  
*Hemerocampa pseudotsugata*  
 MAURO E. MARTIGNONI,<sup>1</sup> PAUL J. IWAI,<sup>1</sup> and LYNFERD J.  
 WICKERHAM  
 (<sup>1</sup>Forestry Sciences Laboratory, USDA, Corvallis, Ore.)  
 J. Invertebr. Pathol. 14(1): 108-110, July 1969

*Candida zeylanoides* has been demonstrated for the first time to be moderately pathogenic for an insect, frequently causing death to larvae when administered in large numbers but rarely lethal in

small numbers. The authors suggest that other closely related species of yeasts should be tested for ability to infect insects. The causative agent was identified at the Northern Division.

- 2537 • **Aflatoxins: Improved Resolution by Thin Layer Chromatography**  
 R. D. STUBBLEFIELD, G. M. SHANNON, and O. L. SHOTWELL  
 J. Ass. Off. Anal. Chem. 52(4): 669-672, July 1969

Water was added to solvent systems for thin-layer chromatography (TLC) of aflatoxins to achieve more reproducible results in laboratories where temperature and relative humidity vary. Resolution of the toxins also improved. Increments of water were added to solvent systems composed of acetone:chloroform (10:90, 12:88, and 15:85, v/v). As the water concentration was increased, separation of aflatoxins B<sub>2</sub> and G<sub>1</sub> improved. These two toxins

are usually the most difficult to resolve in these solvent systems. Separations were the best with water:acetone:chloroform (1.5:12:88, v/v/v). Water added to methanol:chloroform (3:97, v/v) improved resolution of this solvent system but not enough for quantitative analysis, or at times, qualitative analysis. The solvent system water:methanol:ether (1:3:96, v/v/v) separated aflatoxins as well as water:acetone:chloroform (1.5:12:88, v/v/v).

- 2538 • **L-Asparaginase Production by *Erwinia aroideae***  
 R. E. PETERSON and A. CIEGLER  
 Appl. Microbiol. 18(1): 64-67, July 1969

Maximum yields of 1,250 IU (international unit)/gram (dry weight of cells) of L-asparaginase were obtained in 8 hours from *Erwinia aroideae* NRRL B-138. Partial purification and concentration of the extracted L-asparaginase yielded a preparation

with an activity of 275 IU/ml. Only one L-asparaginase was present as determined by electrophoresis, and the enzyme exhibited a pH optimum of 7.5 and K<sub>m</sub> of 3X10<sup>3</sup> M.

- 2539 • **Tremorgenic Toxin from *Penicillium palitans***  
 ALEX CIEGLER  
 Appl. Microbiol. 18(1): 128-129, July 1969

*Penicillium palitans* was isolated from a moldy feed that was implicated in the deaths of some dairy

cows. The culture produced a tremorgen that caused sustained trembling, convulsions, and death in mice.

- 2540 • **Preparation of (Carboxymethyl) cyclohexyldimethylammonium Chloride Hydrazide**  
 T. A. McGUIRE and C. L. MEHLTRETTER  
 J. Med. Chem. 12(4): 727, July 1969

The new compound (carboxymethyl)cyclohexyldimethylammonium chloride hydrazide was prepared by reaction of *N,N*-dimethylcyclohexylamine with

chloroacetate, followed by hydrazide formation with hydrazine.

**2541 • Water-Soluble Films from Partially Acetylated High-Amylose Corn Starch**

A. M. MARK and C. L. MEHLTRETTER  
Staerke 21(4): 92-96. April 1969

Films that were almost completely soluble in hot and cold water were prepared from hot aqueous dispersions of high-amylose corn starch (70% apparent amylose) that had been acetylated in granule form to degree of substitution (D.S.) 0.25 and 0.31 and then subjected to granule disintegration.

Of the methods investigated to disintegrate granules and thereby produce films of high quality, steam jet-cooking of the acetate at 177° C. gave the best results. Films prepared from such dispersions, particu-

larly after clarification, had good tensile strength, high gloss, and excellent clarity. About a tenfold increase in flexibility was also achieved over that of film from untreated acetate. Solubility in hot and cold water, no measurable oxygen transmission, good grease barrier properties, and potential edibility suggest usefulness of these films in packaging dry foodstuffs intended to be added to hot or cold water before use. Other applications are in the protective coating of easily oxidized food products and as sizings for paper and textiles.

**2542 • Heat Denaturation of Soybean 11S Protein**

W. J. WOLF and T. TAMURA  
Cereal Chem. 46(4): 331-344. July 1969

Factors influencing heat denaturation of soybean 11S protein were studied. Effects of time of heating at 100° C. in pH 7.6 solution were determined at different ionic strengths with varying levels of mercaptoethanol. Quantity of precipitate formed and composition of the fraction remaining soluble were determined by ultracentrifugal analysis. Heating at 0.5 ionic strength rapidly converted the 11S protein into a fast-sedimenting aggregate and a slow-sedimenting fraction of 4S. On continued heating, the soluble aggregate increased in size and precipitated. Heating the 11S protein in 0.01 and 0.5 *M* mercaptoethanol accelerated the precipitation reaction. Lowering the ionic strength from 0.5 to 0.1 and to 0.01 in 0.01

*M* mercaptoethanol accelerated disappearance of the 11S protein and formation of the precipitate. When the 11S protein was heated in 0.5 ionic strength buffer containing 0.01 *M* *N*-ethylmaleimide, a 3S fraction and a soluble aggregate formed. The aggregate did not precipitate on continued heating. Heating apparently disrupts the quaternary structure of the 11S protein with subsequent separation of the subunits into two distinct fractions. One fraction consists of soluble subunits of 3-4S, which are stable to heating for 30 minutes or more. The other fraction of subunits has a pronounced tendency to interact to form a soluble aggregate which, in turn, rapidly converts to an insoluble state on continued heating when sulfhydryl groups are present.

**2543 • Crystalline Saponins from Soybean Protein**

A. C. ELDRIDGE and W. J. WOLF  
Cereal Chem. 46(4): 344-349. July 1969

Isolated soybean protein contains saponins which can be removed by a mild acid treatment and crystallized. The crystalline material is a mixture of at least three saponins. Hydrolysis of the saponins reveals soyasapogenols B, C, and D; soyasapogenol B is present in the highest concentration. According to

paper and gas-liquid chromatography the crystalline saponins contain at least five different sugars: glucose, galactose, arabinose, rhamnose, and glucuronic acid. It is yet to be established whether or not the saponins are covalently bonded to the protein.

- 2544 • **Isolation and Chemical Composition of Protein Bodies and Matrix Proteins in Corn Endosperm**  
D. D. CHRISTIANSON, H. C. NIELSEN, U. KHOO, M. J. WOLF,  
and J. S. WALL  
Cereal Chem. 46(4): 372-381. July 1969

Microscopic examination of endosperm cells reveals the presence of starch granules and protein bodies held together by a protein matrix. To separate these subcellular structures, endosperm tissue, obtained 24 days after pollination, was gently homogenized in phosphate buffer. The particulate cell components were then separated by zonal sedimentation on a sucrose density gradient. Sequential fractions were taken from different depths of the gradient tube, analyzed for protein, and examined microscopically. Proteins were characterized by their amino acid composition and starch-gel electrophoretic patterns. Comparisons were made with proteins extracted from endosperm meal with different solvents.

The slowest sedimenting protein zone contained typical albumins and globulins soluble in centrifuga-

tion buffer. A second zone contained a high-molecular-weight protein soluble only in alkali. The amino acid profile of this glutelin protein differed significantly from glutelin extracted from meal with alkali. The third zone contained particles microscopically identical to free protein bodies. The protein in these bodies was characterized as zein. The fourth and fastest sedimenting zone was identified as protein bodies surrounded by a protein matrix. The matrix protein, which resembled glutelin protein in its solubility properties, differs significantly from the extracted glutelin in amino acid composition. The electrophoretic patterns of the reduced and alkylated proteins of zone II and IV, when in combination, resemble the electrophoretic patterns of reduced and alkylated glutelin obtained by direct extraction of endosperm meal.

- 2545 • **An Antibiotic Complex from *Alternaria brassicicola***  
A. CIEGLER and L. A. LINDENFELSER  
Experientia 25(7): 719-720. July 1969

*Alternaria brassicicola* produces an antibiotic complex from which one compound was isolated and chemically characterized. The isolated compound,

named brassicicolin A, is active against yeasts and some pathogenic fungi.

- 2546 • **Composition and Structure of Sorghum Grains**  
J. S. WALL and C. W. BLESSIN  
Cereal Sci. Today 14(8): 264-266, 268-270, 276. August 1969

Some of the yellow-endosperm sorghum introductions have large, hard, easily milled kernels and are devoid of undesirable pigments. Perhaps the greatest advantage of these grains will be in wet- and dry-milling processes where they should produce superior products. Sorghum grain quality is con-

stantly being improved by the plant breeder. New varieties are under development with characteristics that will be more satisfactory for food and industrial uses. As we learn more about sorghum, better methods of milling and processing will be developed. This grain shows great promise for the future.



**2547 • Enzymatic Lysis of Vegetative Cells of *Bacillus popilliae* and Other Sporeformers**

G. R. HRUBANT and R. A. RHODES

Can. J. Microbiol. 15(8): 827-833. August 1969

Spores of *Bacillus popilliae* were isolated from a mixture of spores and vegetative cells by enzymatic digestion of the vegetative cells. Infectivity of the enzyme-treated spores was not affected and germination was enhanced. The enzyme complex, produced extracellularly by an aerobic sporeformer, also induced spheroplasts of *B. popilliae*. Vegetative cells

of bacteria in 13 of 23 genera were lysed by the enzyme, but sporulating forms of all sporeformers tested were not. Consequently, the enzyme can be used for the discriminate selection of sporangial stages of sporulation by the specific lysis of vegetative forms.

**2548\* • Research on Improved Methods of Milling Sorghum Grain**

ROY A. ANDERSON

Proc. 6th Grain Sorghum Research and Utilization Conf.,  
Amarillo, Texas, pp. 32-36. March 5-7, 1969

A number of different conventional and experimental machines can be used to produce grain sorghum grits that meet commercial specifications for brewers' grits. Whole grain sorghum can be roller milled to give a flour containing less than 1% fat and 0.5% ash, with a yield of 68 to 70%. Prepeeling grain sorghum in water can remove essentially all the hull from the kernel. When the peeled grain is impacted, grits with fat contents less than 0.5% can be obtained.

Roller milling of peeled grain sorghum yielded a flour product containing 0.9% fat and 0.3% ash at an extraction of 80%, and an oil-rich fraction having almost 20% fat. By the use of proper conditions, equipment, and procedures, high-quality endosperm fractions can be recovered from grain sorghum. Choice of the milling procedure is governed by the end products desired.

**2549 • Oilseed Protein Sources and Potentials: Soybeans**

R. J. Dimler

Chem. Eng. Progr., Symp. Ser. 65(93): 22-29. 1969

Among the oilseeds, the soybean stands out as a protein source because it is available in the largest amounts (around one-third of the total world supply of oilseeds). In addition, the technology of utilizing soybeans to provide food protein is well advanced. The present status of the supply of soybeans is

briefly reviewed, and the uses of soybean protein are indicated. Some details are given on process development studies at the Northern Division, in which essentially the entire bean is used as a food product, mainly in the form of full-fat soy flour.

- 2550 • **Methylation of Fatty Acids in a Methionine-Dependent *Agrobacterium tumefaciens* Controlled with Exogenous Methionine**  
 TSUNEO KANESHIRO and PAUL J. THOMAS  
 Biochim. Biophys. Acta 187(1): 26-35. July 1969

$^{14}\text{C}$ -transmethylation to form the cyclopropane and branched-chain fatty acids occurred linearly without a lag if exogenous  $[\text{Me-}^{14}\text{C}]$  methionine was pulsed late in the stationary growth phase. With chemostatic cultures (specific dilution rate of  $0.03\text{ h}^{-1}$ ) of a methionine-dependent *Agrobacterium tumefaciens* (strain WM-11), synthesis of only the cyclopropanes was controlled by an excess of exogenous methionine with a restricting concentration of sucrose. When exogenous methionine was a limiting growth factor, cyclopropane fatty acids were not formed and monounsaturated fatty acids accumulated

to a maximal amount. A methyl-branched fatty acid presumed to be  $[\text{C-}^{14}\text{C}]$  methylstearic acid ( $\text{Me-C}_{18}$ ) was also found when excess methionine was present in batch cultures with low aeration. In addition to the specific  $^{14}\text{C}$ -methylation, the "methylstearic" acid was identical in its gas-liquid chromatographic property to a reference  $\text{Me-C}_{18}$  compound. The reference  $\text{Me-C}_{18}$  was prepared from the  $\text{PtO}_2$ -hydrogenation of  $\text{C}_{19}$  cyclopropane acid (dihydrosterculic). Since the "methylstearic" acid was detected in aged cells of static cultures, a biological transmethylation-hydrogenation was suggested.

- 2551 • **Separation of Some Sized and Aspirated Corn Degerminator Fractions by Water Flotation**  
 L. A. WEINECKE and R. R. MONTGOMERY  
 Northwest. Miller 276(7): 14, 16, 18. July 1969

Sized, aspirated corn degerminator streams in the -3-1/2+25 screen size range containing 0.52 to 9.3% oil were separated in a 4-inch diameter water flota-

tion column, yielding grit fractions of from 0.2 to 0.8% oil and germ fractions of 8 to 32% oil on a moisture-free basis.

- 2552 • **Polyesteramides from Linseed and Soybean Oils for Protective Coatings: Diisocyanate-Modified Polymers**  
 L. E. GAST, WILMA J. SCHNEIDER, G. E. McMANIS, and J. C. COWAN  
 J. Amer. Oil Chem. Soc. 46(7): 360-364. July 1969

New polymeric coating materials have been prepared by a triethylenediamine-catalyzed reaction of hydroxyl-terminated polyesteramides (HTPA) from soybean or linseed oils with diisocyanates. Eight dibasic acids or anhydrides were reacted with excess *N,N*-bis(2-hydroxyethyl) fatty amide to yield HTPA; those containing 10 mole percent excess gave isocyanate-modified polymers with best overall film properties.

Reactivity of four diisocyanates with a linseed-HTPA was measured by disappearance of the isocyanate band in the infrared.

Depending on chemical composition, structure, and

curing conditions, films prepared from these polymers have a wide range of drying characteristics, hardness, and chemical resistance. Drying times of linseed HTPA-urethane polymer films varied from 0.3 to 48 hours, hardness values (Sward) were from 4 to 70, alkali resistance ranged from 2 to 126 minutes, and the hydrochloric acid and xylene resistances were good to excellent. Impact resistance exceeded 160 in.-lb. for all films except two. The soybean-derived polymer films likewise exhibited a wide range of properties; they chiefly differed from linseed-derived films in having greater flexibility and improved alkali resistance.

**2553 • Structure of Unsaturated Vegetable Oil Glycerides: Direct Calculation from Fatty Acid Composition**

C. D. EVANS, D. G. McCONNELL, G. R. LIST, and  
J. C. COWAN

J. Amer. Oil Chem. Soc. 46(8): 421-424. August 1969

Composition and structure of unsaturated glycerides of vegetable oils can be calculated directly from the fatty acid composition of the oil. Fatty acid distribution on the 2 position as normally determined by lipase hydrolysis is calculated from the composition of the whole oil by applying the following three rules in order: (1) Saturated fatty acids and those with chain length greater than 18 carbons are first distributed equally and randomly on the 1 and 3 positions of the glycerol moiety; (2) oleic and linolenic acids are treated equally, or as a unit, and distributed

equally and randomly on all three glyceride positions with any excess from the 1 and 3 positions being added to the 2 position; and (3) all remaining positions are filled by linoleic acid. Remarkably good agreement between the calculated and experimentally determined fatty acid distributions is shown for soybean, linseed, safflower, and many other vegetable oils whose compositions are reported in the literature. An association between oleic and linolenic acid within the glyceride structure of some vegetable oils is evident.

**2554 • Composition of Oilseeds. A List of Publications for 1968**

NORTH. UTIL. RES. DEVELOP. DIV.

U.S. Agr. Res. Serv., ARS-71-23-7, 4 pp. August 1969. [Processed]

**2555 • Processing Oilseeds, Oil, and Meal. A List of Publications and Patents for 1968**

NORTH. UTIL. RES. DEVELOP. DIV.

U.S. Agr. Res. Serv., ARS-71-24-7, 2 pp. August 1969. [Processed]

**2556 • Edible Soybean Oil. A List of Publications for 1968**

NORTH. UTIL. RES. DEVELOP. DIV.

U. S. Agr. Res. Serv., ARS-71-25-7, 3 pp. August 1969. [Processed]

**2557 • Edible Soybean Protein Products. A List of Publications For 1968**

NORTH. UTIL. RES. DEVELOP. DIV.

U. S. Agr. Res. Serv., ARS-71-26-7, 3 pp. August 1969. [Processed]

**2558 • Chemically Modified Oil Products and Industrial Uses. A List of Publications and Patents for 1968**

NORTH. UTIL. RES. DEVELOP. DIV.

U.S. Agr. Res. Serv., ARS-71-27-7, 4 pp. August 1969. [Processed]

**2559 • Review Articles on Oilseed Crops Research. A List of Publications for 1968**

NORTH. UTIL. RES. DEVELOP. DIV.

U.S. Agr. Res. Serv., ARS-71-29-7, 2 pp. August 1969. [Processed]



- 2560 • **Amadori Compounds as Nonvolatile Flavor Precursors in Processed Foods**  
FRANK D. MILLS, B. GENE BAKER, and JOHN E. HODGE  
J. Agr. Food Chem. 17(4): 723-727. July-August 1969

Amadori compounds, derived from the condensation of amino acids with reducing sugars in the Maillard reaction, have been isolated from browned, dehydrated fruits and vegetables, bakery products, cane and beet molasses, and cured tobacco. The non-volatile compounds in their enolic forms are precursors of flavor compounds, particularly those with burnt and caramel-like aromas. A model basic Amadori compound, 1-deoxy-1-piperidino-D-fructose, was pyrolyzed and the volatile products were isolated.

These products, high boiling liquids and sublimable solids, consisted mainly of 4-carbon methyl reductones,  $\alpha$ -piperidino- $\gamma$ -butyrolactone, and piperidine amides of carbonic, formic, acetic, butyric, glycolic, and lactic acids. Only a trace of *N*-lactylpiperidine was found, indicating a predominance of 4:2 over 3:3 splitting of the hexose moiety. A reaction course through the 4-carbon reductones to the 2- and 1-carbon acid amides is suggested.

- 2561 • **An Orange-Yellow Mutant of *Aspergillus parasiticus* Produces Aflatoxin**  
J. J. ELLIS  
Mycologia 61(3): 651-653. May-June 1969

A natural color mutant originated from the strain on which the species description of *Aspergillus parasiticus* Speare was based. Colonies of the new mutant remain a distinct shade of orange yellow as

they age. When grown on rice, the mutant produced nearly twice the amount of aflatoxin B<sub>1</sub> and G<sub>1</sub> as the parent strain.

- 2562 • **2-Oxazolidinone Derivatives of D-Glucose and Glycoaldehyde**  
W. E. DICK, JR., D. WEISLEDER, and J. E. HODGE  
J. Org. Chem. 34(9): 2654-2660. September 1969

A 4-hydroxy-3-phenyl-5-(D-*arabino*-1,2,3,4-tetrahydroxybutyl)-2-oxazolidinone structure (III) is established for the compound previously registered as 2-carbanilino-D-glucose. Hydrolysis of *N*-(2-*O*-phenylcarbamoyl- $\beta$ -D-glucopyranosyl)piperidine first forms 2-*O*-phenylcarbamoyl-D-glucopyranose which rapidly converts into III in alkaline solution. The mechanism proposed for this cyclization requires attack of the amido nitrogen on the adjacent carbonyl group.

Cyclization of glycoaldehyde carbanilate to 4-hydroxy-3-phenyl-2-oxazolidinone in high yield at pH 4 requires a free carbonyl group. Treatment of III with methanolic hydrogen chloride produces an  $\alpha$ -D-glucofurano-2-oxazolidinone derivative, 5-(D-*glycero*-1, 2-dihydroxyethyl)tetrahydro-6-hydroxy-3-phenylfuro [2,3-*d*] oxazol-2-(3*H*)-one, isolated as a triacetate. The structure is assigned by nmr analysis.

**2563 • Olefin-Tetracyanoethylene Oxide Adducts and Some of Their Derivatives**

M. O. BAGBY, C. R. SMITH, JR., and I. A. WOLFF

J. Org. Chem. 34(9): 2732-2735. September 1969

The reaction of tetracyanoethylene oxide with methyl erucate (methyl *cis*-13-docosenoate) and methyl brassidate (methyl *trans*-13-docosenoate) gave 2,2,5,5-tetracyano-3-(11-carbomethoxydodecyl)-4-octyltetrahydrofurans in good yields. In methanol containing 5% hydrogen chloride, both addition products undergo partial and selective methanolysis to give 2,5-dicyano-2,5-dicarbomethoxytetrahydrofuran

derivatives. The newly formed carbomethoxy groups are *trans* to their adjacent alkyl groups. Both addition products also are partially and selectively hydrolyzed on the surface of silica gel to give 2,5-dicyano-2,5-dicarbamoyltetrahydrofuran derivatives in good yields. The newly formed carbamoyl groups have a *cis* relationship to each other.

**2564 • Kinetic Rate Constants Determined by a Digital Computer**

R. O. BUTTERFIELD

J. Amer. Oil Chem. Soc. 46(9): 429-431. September 1969

The difficulty of determining rate constants for complex reactions has been overcome with the development of a general digital computer program that can determine up to 10 rate constants in any reaction scheme which can contain as many as 10 components. A given reaction to which the experimental data are to be fit is described to the computer in a short one-step integration subprogram,

which solves the differential equations representing the scheme. Only this subprogram needs to be rewritten to change the reaction scheme. Special features of the program and length of calculations are discussed. A copper-chromite hydrogenation of a mixture of linolenate and conjugated linoleate demonstrates what the program does and what is a typical output.

**2565 • A Bibliography on the Solvent Extraction of Soybeans and Soybean Products 1944-1968**

A. C. ELDRIDGE

J. Amer. Oil Chem. Soc. 46(9): 458A, 460A, 462A,

464A, 496A, 498A, 500A, 502A. September 1969

This bibliography covers the effect of several solvents on oil removal, toxicity, color, texture or flavor of soybean flakes, flour and protein. The lit-

erature search included processes, principles and apparatus.

**2566 • Some Radiochemical Experiments on Minor Constituents in Soybean Oil**

T. L. MOUNTS, C. D. EVANS, H. J. DUTTON, and J. C. COWAN

J. Amer. Oil Chem. Soc. 46(9): 482-484. September 1969

A  $^{14}\text{C}$ -labeled high-molecular-weight hydrocarbon and an insecticidal compound were added as minor constituents in soybean oil samples. Liquid scintillation counting was used to assay the radioactivity of the oil preceding and after laboratory simulations of commercial processing procedures (bleaching and deodorization). Radiochemical techniques were found to be highly sensitive and quantitative and detection was unaffected by chemical modification or decomposition of the parent com-

pound. Labeled ( $^{14}\text{C}$ ) benzo( $\alpha$ )pyrene was retained primarily by the oil during extraction, filtering, solvent stripping, deodorization, and treatment with AOCS bleaching earths. Treatment of the oil with activated charcoal effected removal of this hydrocarbon. Bleaching was ineffective in removing added  $^{14}\text{C}$ -endrin from the oil but a deodorization using specific conditions of temperature ( $250^\circ\text{C}$ .), time (2 hours), and pressure (4.5 mm.) removed this constituent.

- 2567 • **Removal of Copper from Hydrogenated Soybean Oil**  
 R. E. BEAL, K. J. MOULTON, H. A. MOSER, and L. T. BLACK  
*J. Amer. Oil Chem. Soc.* 46(9): 498-500. September 1969

Hydrogenation with a copper-chromite catalyst at 170° C., 30 p.s.i., increased the copper content of a refined, bleached soybean oil from 0.02 to as much as 3.8 p.p.m. Removing residual copper from soybean oil is essential to the successful use of copper catalysts for selective hydrogenation. Various methods were examined to remove this copper, including alkali refining, bleaching, acid washing, citric acid treatment, and cation-exchange resin treatment. Properly conducted, each of the methods except alkali refining gives 95% or higher removal of copper introduced during hydrogenation. Ion exchange

appears to be the most economical, but addition of about 0.01% citric acid during deodorization may be needed to inactivate traces of unremoved copper. Soybean oil hydrogenated with a copper-chromite catalyst, bleached or treated with an ion-exchange resin, and deodorized with 0.01% citric acid added had low AOM peroxide values and acceptable flavor scores after eight days at 60° C. which indicate that removal of residual copper from the oil should be adequate for the production of stable oils low in linolenic acid content.

- 2568 • **Edible Oil Quality as Measured by Thermal Release of Pentane**  
 C. D. EVANS, G. R. LIST, R. L. HOFFMANN, and HELEN A. MOSER  
*J. Amer. Oil Chem. Soc.* 46(9): 501-504. September 1969

As part of their thermal decomposition products, fatty hydroperoxides produce normal hydrocarbons. The extent of hydrocarbon formation can be measured and associated with the quality and potential stability of an oil. Edible oils containing linoleic acid develop 13-hydroperoxy-9,11-octadecadienoic acid as one product of autoxidation. On thermal decomposition this hydroperoxide yields pentane; the amount released has been correlated with the

flavor scores of fresh and aged soybean and cottonseed oils and with the peroxide values of these oils. The quantity of pentane released has an inverse linear relationship to flavor score and a direct linear relationship to peroxide values. Edible oils exposed to light exhibit a different relationship between flavor score and thermally derived pentane than do the same oils when autoxidized in the dark.

- 2569 • **Wax Esters from Sunflower Oil Tank Settlings**  
 R. KLEIMAN, F. R. EARLE, and I. A. WOLFF  
*J. Amer. Oil Chem. Soc.* 46(9): 505. September 1969

Gas chromatographic analysis of wax esters isolated from sediment found in a sunflower oil tank revealed components containing 42 to 60 carbon atoms, with those containing 44 and 46 the most prominent. The acids ranged from C<sub>14</sub> to C<sub>30</sub> (44% C<sub>20</sub> and 22% C<sub>22</sub>) and alcohols from C<sub>18</sub> to C<sub>34</sub> (32% C<sub>24</sub> and 28% C<sub>26</sub>). Wax esters isolated

from sunflower hull lipids were similar to those from the tank settlings. This similarity suggests that the wax esters in the tank settling originated in the sunflower seed hull.

The wax ester composition calculated from a random combination of alcohols and acids from the tank settling agrees well with the composition found.



- 2570** • **Cationic Aminoethyl Cereal Flours as Wet-End Paper Additives**  
H. D. HEATH, A. J. ERNST, B. T. HOFREITER, and J. C. RANKIN  
Tappi 52(9): 1647-1651. September 1969

Corn, wheat, and sorghum flours and a corn starch were allowed to react in a semidry process with ethylenimine. Avoidance of water as a reaction medium offers economic advantages. The aminoethyl reaction products were evaluated in wet-end additions on a pilot fourdrinier in the production of filled

bond and bag papers. These cationic derivatives improved strength and clay retention as much as high-quality commercial cationic starch. Performance was also satisfactory with respect to paper machine operability, white water characteristics, and paper brightness and color.

- 2571** • **Producing Quality Sorghum Flour on Wheat Milling Equipment**  
ROY A. ANDERSON  
Northwest. Miller 276(10): 10-12, 14-15. October 1969

Operating conditions were established for obtaining a quality sorghum flour by the milling of whole grain sorghum on the Buhler experimental flour mill. This mill has been historically used for testing the millability of wheat. When grain sorghum was tempered

to 19 to 19.5% moisture, flour was obtained at about 68% extraction containing 1% of fat or less, and less than 0.5% ash. Several other samples of sorghum were milled with similar results.

- 2572** • **Urethane Plastics Based on Starch-Derived Glycosides**  
F. H. OTEY, R. P. WESTHOFF, W. F. KOWLEK,<sup>1</sup>  
C. L. MEHLTRETTER, and C. E. RIST  
(<sup>1</sup>USDA Biometrical Serv., Peoria, Ill.)  
Ind. Eng. Chem., Prod. Res. Develop. 8(3): 267-274. September 1969

Alcoholysis of castor oil with starch-derived glycosides yields polyols that can be used in preparing urethane plastics. Equivalent weights can be varied from 200 to 336, depending upon the ratio of glycoside and castor oil used. As the polyol equivalent weight is decreased, the derived plastics become stronger, harder, and more resistant to heat. Incorporation of up to 60% starch as a reactive filler into the plastic formulations further improves these properties generally; but its effect is even more

pronounced when the plastics are made with high equivalent weight polyols. Properties are usually the best when the NCO/OH ratio is 1.5 or greater.

A wide range of physical properties were built into the plastics by properly selecting the polyol and the amounts of isocyanate and starch. The range of properties these relatively inexpensive materials provide suggests that starch and starch-derived polyols have potential for the commercial production of urethane plastics.

**2573 • Effect of Temperature and Concentration on the Chlorohydration of Allyltrimethylammonium Chloride**

C. L. MEHLTRETTER, T. A. McGUIRE, C. A. GLASS, and  
C. A. WILHAM

Ind. Eng. Chem., Prod. Res. Develop. 8(3): 279-281.

September 1969

Reaction of allyltrimethylammonium chloride with chlorine in aqueous solution in the temperature range of 25° to 32° C. gave a nearly quantitative yield of a 3 to 2 mixture of *N*-(2-chloro-3-hydroxypropyl)- and *N*-(3-chloro-2-hydroxypropyl) trimethylammonium chlorides. Chlorohydration at 42° to 46° C. formed an appreciable quantity of the chlorination product, *N*-(2,3-dichloropropyl)-trimethylammonium chloride.

A change in concentration of allyltrimethylammonium chloride from 15 to 35% did not affect the course of chlorohydration at lower temperatures. Proton magnetic resonance spectra established the structure and composition of the products. The improved synthesis should allow more efficient production of cationic starches and ion exchange cellulose.

**2574 • Relationship of Amino Acid Composition and Wheat Protein Properties**

L. H. KRULL and J. S. WALL

Bakers Dig. 43(4): 30-34, 36, 38-39. August 1969

The relationship is shown between properties of wheat gluten and amino acid composition. The unique cohesive properties of the gluten protein are due to intermolecular interactions between amino acid functional groups present in the protein chains. Although single hydrogen bonds or hydrophobic bonds may be relatively weak, when multiplied by the vast number possible in wheat gluten, these forces are effective in producing cohesion. Salt and pH produce electrostatic effects between protein molecules. The

high molecular weights of the glutenin proteins resulting from interchain disulfide bonding further increase the number of sites for association and contribute to the elastic quality of the gluten. Agents effective in modifying gluten protein behavior in solution also alter the flour doughs. Fundamental research on the influence of additives to properties of gluten proteins may provide information needed to adapt flours better to new processing techniques.

**2575 • Copolymers of Modified Starches with Polyacrylonitrile**

GEORGE F. FANTA, ROBERT C. BURR, C. R. RUSSELL, and  
C. E. RIST

J. Polym. Sci., Pt. A-1, 7(7): 1675-1681. July 1969

A study was made of the ceric ammonium nitrate-initiated graft polymerization of acrylonitrile (AN) onto a number of modified starches that had been reduced in molecular weight by either acid, hypochlorite, or enzyme treatment. With highly soluble starches, much of the starting material was recovered as ungrafted carbohydrate, and the reaction product was largely dimethylformamide-soluble polymer with a high polyacrylonitrile (PAN) content. The molecular weight of grafted PAN was lower when the modified starches existed as granules in water dispersion; however, heating (60° C.) an aqueous slurry of an acid-modified corn starch (with intact granules) before the reaction had rela-

tively little effect on the composition of the copolymer. Decreasing the concentrations in water of modified starch and AN resulted in more frequent and lower molecular weight grafts of PAN. Aqueous methanol as a reaction medium for an acid-modified starch with intact granules led to more frequent grafting of lower molecular weight PAN than when water alone was used. The number of grafted chains, however, was fewer than found with unmodified wheat starch under comparable conditions. A modified starch with the granule structure completely broken down gave no detectable reaction in aqueous methanol.

- 2576 • **Scanning Electron and Phase-Contrast Microscopy of Bacterial Spores**  
 L. A. BULLA, G. ST. JULIAN, R. A. RHODES, and  
 C. W. HESSELTINE  
 Appl. Microbiol. 18(3): 490-495. September 1969

The three-dimensional images of free and intra-sporangial spores produced by scanning electron microscopy show surface structures not visible by phase-contrast microscopy. Although fine surface detail is not elucidated by scanning electron microscopy, this technique does afford a definitive picture

of the general shape of spores. Spores of *Bacillus popilliae*, *B. lentimorbus*, *B. thuringiensis*, *B. alvei*, *B. cerius*, and *Sarcina ureae* have varying patterns of surface ridge formation, whereas spores of *B. larvae*, *B. subtilis*, and *B. licheniformis* have relatively smooth surfaces.

- 2577 • **Dihydrosterculic Acid, A Major Fatty Acid Component of *Euphoria longana* Seed Oil**  
 R. KLEIMAN, F. R. EARLE, and I. A. WOLFF  
 Lipids 4(5): 317-320. September 1969

The seed oil of *Euphoria longana*, Sapindaceae, contains 17.4% of 9,10-methyleneoctadecanoic (dihydrosterculic) acid. This identification is based on information from thin-layer chromatography (TLC), infrared analysis, gas-liquid chromatography (GLC), nuclear magnetic resonance, and mass spectroscopy. Since GLC of the oil showed components that emerged between the usual triglycerides, the cyclopropanoid acid is apparently a triglyceride constituent. The

presence of smaller amounts, less than 1%, of cyclopropanoid fatty acids of different chain lengths is indicated by GLC and TLC analyses of the methyl esters. The other major fatty acids in this oil are: 16:0 (19%), 18:0 (7%), 18:1 (36%), 18:2 (6%), 18:3 (5%), and 20:0 (4%). *Euphoria* oil contains considerably larger amounts of cyclopropanoid fatty acids than previously reported in other seed oils.

- 2578 • **Microbial Hydration of *Cis*-9-Alkenoic Acids**  
 E. N. DAVIS, L. L. WALLEN, J. C. GOODWIN, W. K. ROHWEDDER,  
 and R. A. RHODES  
 Lipids 4(5): 356-362. September 1969

The activity of a *cis*-9-fatty acid hydratase produced by a *Pseudomonas* sp. (NRRL B-3266) isolated from soil was compared with that of another isolate previously reported (NRRL B-2994). The presence of appropriate fatty acids for at least 4 hours during aerobic growth in yeast extract medium increased subsequent enzyme activity. Such cells anaerobically hydrated several *cis*-9-alkenoic acids to 10-hydroxy fatty acids and aerobically formed 10-keto acids, which were partially degraded to shorter chain keto acids. Melting point, gas chromatography, infrared, mass spectrometry, and optical rotatory

dispersion data are given. Six fatty acids having *cis*-9-unsaturation produced hydrated products, but several enoic acids having *trans*-9-unsaturation or double bonds in other than the 9-position were inactive as substrates. The (-)-10-hydroxypalmitic acid produced from palmitoleic acid is considered to have the  $D$  configuration. Yields of 71% crude crystalline product from 15 g. of oleic acid and 53% from 11 g. of palmitoleic acid were obtained in 5-liter anaerobic fermentations with NRRL B-3266. Methyl esters, triolein, and oleyl alcohol were not hydrated.



**2579 • Reagent for Detection of S- $\beta$ -Aminoethylcysteine**

JOHN A. ROTHFUS

Anal. Biochem. 30(2): 279-283. August 1969

S- $\beta$ -Aminoethylcysteine(AE-Cys) was distinguished from other amino acids on paper by spraying with *p*-dimethylaminobenzaldehyde in aqueous *N,N*-dimethylformamide (DMF) and heating at 110° C. to produce a stable orange product. As little as 0.005  $\mu$ mole of AE-Cys was detected by this treatment. The colored product, which gave positive reactions

with ninhydrin and iodine-azide solution, had absorption maxima at 390 and 465  $\mu$  in DMF and followed Beer's law for levels of AE-Cys from 0.1 to 0.7  $\mu$ mole. This color reaction has been useful in purifying AE-Cys peptides from enzymic digests of wheat gluten proteins.

**2580 • Computer Program to Convert Spectroreflectometric Data into Color Designations**

GEORGE E. McMANIS and LYLE E. GAST

J. Paint Technol. 41(537): 581-582. October 1969

A computer program has been written that converts reflectance values of a test sample, obtained from a spectrophotometer, into CIE color designation values and that computes the color difference

in NBS units from a standard. This program is particularly helpful for paints with high reflectance values such as white paints.

**2581 • Food Uses of Soybeans. Research at the Northern Laboratory**

HOWARD M. TEETER and WILBUR C. SCHAEFER

Soybean Dig. 30(1): 16-18. November 1969

Continued growth is predicted in both domestic and foreign food use of soybeans. Past contributions and current research of the Northern Division include studies on flavor stability of soybean oil, flavor and other components of soybean meal and protein, Oriental foods from soybeans, the "Village Process"

for making full-fat soybean flour, and corn-soya-milk (CSM) blend. Future research is expected to emphasize flavor, functional, and physiological properties of edible soybean products; and work will be continued on full-fat soybean flour and food products similar to CSM.

**2582 • Oilseed Proteins**

R. J. DIMLER

Chem. Eng. Progr. 65(9): 20-26. September 1969

Oilseeds have potential for increased use as sources of food proteins. Generally, processing is needed to improve food value or acceptability, or both. Factors of importance to the user include

protein concentration, flavor, functional properties, and nutritional quality. Some engineering research studies on soybeans and cottonseed are reviewed in these terms.

- 2583 • **Base-Hydrolyzed Starch-Polyacrylonitrile (S-PAN) Graft Copolymer. S-PAN-1:1, PAN M.W. 794,000**  
L. A. GUGLIEMELLI, M. O. WEAVER, C. R. RUSSELL,  
and C. E. RIST  
J. Appl. Polym. Sci. 13(9): 2007-2017. September 1969

Starch-polyacrylonitrile (S-PAN) graft copolymer consisting of one part starch and one part polyacrylonitrile (M.W. 794,000) was hydrolyzed with aqueous KOH at 80° and 100° C. to polyelectrolytes containing carboxyl and amide functionality but void of infrared-detectable amounts of nitrile. Viscosities of potassium salts of the polyelectrolytes in water were pH dependent with peak viscosities at 8.5. These dispersions were characterized by unusually high

viscosities, 15,000 to 26,500 cp. at 1% concentration, and by the ability to retain significant viscosities, 300 to 950 cp., upon dilution to 0.03% concentrations. A 1% dispersion of hydrolyzed S-PAN had a viscosity of 5,000 cp. in the presence of 8% KCl; however, the depressing effect of added salts on viscosity of aqueous dispersions of hydrolyzed S-PAN increases as the valence of the cation increases.

- 2584 • **Rapid Micromethod for Location of Ene-yne and  $\alpha$ -Hydroxy Conjugated Diene Systems in Straight-Chain Compounds**  
G. F. SPENCER, R. KLEIMAN, F. R. EARLE, and I. A. WOLFF  
Anal. Chem. 41(13): 1874-1876. November 1969

For unsaturated aliphatic compounds with known functional groups, oxidative cleavage at the sites of unsaturation and identification of the fragments formed constitute a convenient means of bond location. Rapid analytical procedures coupling ozonolysis and gas chromatography have been developed. Such methods have generally been restricted to locating double bonds. Triple bonds have been located by

ozonolysis in acidic media, but recovery and identification of the acids produced are time consuming. The procedure described is rapid, facile, specific for double and triple bonds, and is applicable to very small samples. The method has a further advantage: It can be used to locate both the bonds and the hydroxyl group in hydroxy conjugated dienes.

- 2585 • **A Novel Spectrophotometric Procedure for Half-Cystine Residues in Proteins**  
MENDEL FRIEDMAN and L. H. KRULL  
Biochem. Biophys. Res. Commun. 37(4): 630-633. November 1969

A spectrophotometric assay for half-cystine residues in proteins was developed based on the following observations: (1) Protein SH groups, generated by reduction of disulfide bonds with mercaptoethanol, are selectively modified by 4-vinylpyridine at pH 7.5 to S- $\beta$ -(4-pyridylethyl)-L-cysteine residues; (2) the molar extinction coefficient of the pyridine ring in

S-pyridylethylproteins is generally lower than the corresponding value in S- $\beta$ -pyridylethylcysteine (PEC); (3) the S-pyridylethylcysteine in the modified proteins is stable to protein acid-hydrolysis conditions; and (4) the molar extinction coefficient of the pyridine ring in hydrolyzed  $\beta$ -pyridylethylproteins is identical to the value found for PEC.

- 2586\* • **The Work at Northern Laboratory**  
G. C. MUSTAKAS  
Soybean Dig. 29(12): 36. September 1969

Soybeans will be a primary source of food that can help alleviate caloric and protein malnutrition in the developing countries. Work at the Northern Regional Research Laboratory has emphasized two engineering processes for making full-fat soy flour, a product

both high in protein and energy value. One process will be applicable in urban communities, whereas the other process will be used in remote villages where steam and electrical power are unavailable.

- 2587 • **Chemical and Physical Properties of Soybean Proteins**  
W. J. WOLF  
Bakers Dig. 43(5): 30-31, 34-37. October 1969

A review of the chemical and physical properties of soybean proteins as related to the use of soybean flours, concentrates, and isolates by the baking industry.

- 2588\* • **Enzymatic Browning of Foods**  
JOHN E. HODGE  
*In* "Current Topics in Food and Nutrition," eds. Margaret O. Osborn and Elizabeth M. Osman, Proc. Home Econ. Workshop, Univ. of Iowa, Iowa City, June 10-28, 1968, pp. 127-146. 1969

Enzymatic and nonenzymatic browning reactions are classified with reference to the oxidative or nonoxidative reactions that occur initially in harvested natural foods. Because hydrolytic enzymes usually participate in so-called nonenzymatic browning reactions to liberate the reactive sugars, peptides, and amino acids from their conjugates, and

because phenolase browning reactions are enzymatic only in the initial steps, this clarification seems necessary. The occurrence, effects, substrates, enzymes, reaction sequence, inhibition, and control of oxidative enzymatic browning are illustrated and discussed in review.

- 2589 • **Low-Fat Endosperm Fractions from Grain Sorghum**  
R. A. ANDERSON, R. R. MONTGOMERY, and L. H. BURBRIDGE  
Cereal Sci. Today 14(11): 366-368. November 1969

Engineering studies on the production of grits and flour from grain sorghum, by a variety of dehulling, degerminating, and milling machines, resulted in the recovery of endosperm fractions in yields of 68 to 75%. Always, the +14 and +20 endosperm frac-

tions had fat contents of less than 1%, a figure that meets commercial specifications for brewers' grits. The fat content of the flour, recovered by roller milling grain sorghum, was less than 1%, and its ash content was 0.5%.

- 2590 • **Roll and Extrusion-Cooking of Grain Sorghum Grits**  
R. A. ANDERSON, H. F. CONWAY, V. F. PFEIFER, and E. L. GRIFFIN, JR.  
Cereal Sci. Today 14(11): 372-375, 381. November 1969

Grain sorghum continues to expand as a major farm crop. However, almost all the grain is used for feeding, and only a small amount finds its way into food and industrial channels. New commercial products will provide outlets for the increasing acreage of this grain. For many industrial and food applications, it is necessary to alter the properties of the endosperm fractions by cooking them to various

degrees of gelatinization. By varying cooking conditions, a wide range of products was achieved that had different water absorptions and solubilities as well as different viscosities of cooked and uncooked suspensions in water. Studies are described on processing of grain sorghum grits by roll- and extrusion-cooking and on investigating such variables as moisture, temperature, and compression ratios.



- 2591 • **Survey of Some *Actinomycetales* for  $\alpha$ -Galactosidase Activity**  
A. J. LYONS, JR., T. G. PRIDHAM, and C. W. HESSELTINE  
Appl. Microbiol. 18(4): 579-583. October 1969

The enzyme  $\alpha$ -galactosidase offers potential to (1) eliminate possibly the flatus-inducing factor(s) in edible beans, (2) eliminate raffinose during beet-sugar processing, and (3) determine raffinose analytically. Accordingly, 20 genera of the order *Actinomycetales* Buchanan 1917 were tested to detect evidence of  $\alpha$ -galactosidase activity. Test filtrates were prepared with a medium containing D-galactose and soybean meal. Enzyme activity was demonstrated through cellulose thin-layer chromatography.

Of 123 strains tested, 28 produced extracellular  $\alpha$ -galactosidase. Almost all were streptomycetes. Members of the genera *Actinoplanes* Couch 1950, *Micromonospora* Orskov 1923, and *Promicromonospora* Krasil'nikov et al. 1961 also exhibited  $\alpha$ -galactosidase activity. Additional tests led to the selection of five strains whose filtrates degraded melibiose, raffinose, and stachyose but not lactose and sucrose. Tests also were made with several soybean preparations.

- 2592 • **Survey of Cereal Grains and Soybeans for the Presence of Aflatoxin: I. Wheat, Grain Sorghum, and Oats**  
ODETTE L. SHOTWELL, C. W. HESSELTINE, H. R. BURMEISTER, W. F. KWOLEK<sup>1</sup>, GAIL M. SHANNON, and H. H. HALL  
(<sup>1</sup>USDA Biometrical Serv., Peoria, Ill.)  
Cereal Chem. 46(5): 446-454. September 1969

A total of 531 wheat samples, 533 grain sorghum samples, and 304 oat samples, representing all marketing grades, were analyzed for the presence of aflatoxin. Samples were extracted by the procedure, slightly modified, developed by the Food and Drug Administration for the analysis of peanut and peanut products. Extracts were assayed for the presence of aflatoxins by thin-layer chromatography (TLC). The sensitivity limit of the analysis as carried out was 2 to 5 p.p.b. of the metabolite. Accord-

ing to results of TLC, two wheat samples, six grain sorghum samples, and three oat samples, all in poorer grades, appeared to contain low levels of aflatoxin (2 to 19 p.p.b.). Samples that were positive by TLC were tested in ducklings. No aflatoxinlike activity was detected in grain sorghum samples. Although the duckling test indicated that traces of toxins might be present in wheat and oat samples, more likely the test was negative. The sensitivity of the test as conducted was 1 to 2 p.p.b.

- 2593 • **Survey of Cereal Grains and Soybeans for the Presence of Aflatoxin. II. Corn and Soybeans**  
ODETTE L. SHOTWELL, C. W. HESSELTINE, H. R. BURMEISTER, W. F. KWOLEK,<sup>1</sup> GAIL M. SHANNON, and H. H. HALL  
(<sup>1</sup>USDA Biometrical Serv., Peoria, Ill.)  
Cereal Chem. 46(5): 454-463. September 1969

A total of 1,311 corn samples and 866 soybean samples, including samples from all grades and two different crop years, 1964 and 1965, were assayed for the presence of aflatoxin. Samples were extracted by the procedure used in the survey of wheat, grain sorghum, and oat samples, and extracts were tested for the presence of aflatoxin by thin-layer chromatography (TLC). The sensitivity limit of the analysis

as carried out was 2 to 5 p.p.b. of the metabolite. Of the 35 corn samples that were positive by TLC, 30 gave aflatoxinlike responses in ducklings. Five of these were in grade 5, and 25 were in sample grade. Two of the 866 soybean samples tested contained aflatoxin by TLC and the duckling test. These were in sample grade.

**2594 • A Note on the Detection of Leucoanthocyanins in Defatted Soybean Flakes**

L. C. WANG, A. K. SMITH, and J. C. COWAN

Cereal Chem. 46(5): 468-470. September 1969

A leucoanthocyanin fraction was isolated from ethanolic extracts of soybean flakes by chromatography on a polyvinylpyrrolidone column. The conventional procedure to convert leucoanthocyanins to

anthocyanidins was found unsuitable with extracts from soybean flakes. Under conditions reported here an anthocyanidin-like derivative that absorbed at 275  $\mu$  was isolated.

**2595 • Polyacrylamide-Gel Electrophoresis of Reduced and Alkylated Soybean Trypsin Inhibitors**

A. C. ELDRIDGE and W. J. WOLF

Cereal Chem. 46(5): 470-478. September 1969

Polyacrylamide-gel electrophoresis of native and reduced-alkylated soybean trypsin inhibitors showed them to be different proteins rather than intramolecular disulfide forms or intermolecular disulfide polymers of a single protein. Reduction alone or reduction followed by alkylation caused one of the

inhibitors to migrate much slower in 8 *M* urea than did the native inhibitor. This decrease in mobility, which was accompanied by an increase in intrinsic viscosity, is attributed to an unfolding after cleavage of the disulfide crosslinkages.

**2596 • Corn Dry-Milling: Cold-Tempering and Degermination of Corn of Various Initial Moisture Contents**

O. L. BREKKE and W. F. KWOLEK<sup>1</sup>

(<sup>1</sup>USDA Biometrical Serv., Peoria, Ill.)

Cereal Chem. 46(5): 545-559. September 1969

Shelled corn initially containing 21, 17, and 13% moisture was cold-tempered (i.e., without heat) and degerminated on a pilot-plant scale to determine the variation in dry-milling response. Corn for the two lower moisture levels had been dried on the ear under extremely mild conditions typically used for seed corn. For the tempering step, low, moderate, and high addition levels were used for the first-temper water; temper times were 0.5 to 4 hours. Upon degermination of tempered corn from each of the three

lots, 21%-moisture corn gave products whose yields and oil contents were excellent. Over-all, this lot had the highest yield of flaking grits with lowest oil content, along with more recoverable oil, and was the most consistent in its milling response. The 17%-moisture corn gave slightly poorer results and the 13%-moisture corn, appreciably poorer. Blending corn of various moisture contents aggravates the dry-miller's problems.

**2597 • Publications and Patents of the Northern Utilization Research and Development Division, January-June 1969**

NORTH. UTIL. RES. DEVELOP. DIV.

U.S. Agr. Res. Serv., Unnumb. Pub., 48 pp. [July 1969]

- 2598 • **Measuring Thickness of Excised Mature Corn Pericarp**  
 M. J. WOLF, IRENE M. CULL, J. L. HELM,<sup>1</sup> and M. S. ZUBER<sup>2</sup>  
 (<sup>1</sup>Department of Agronomy, and <sup>2</sup>USDA Crops Research Div.,  
 University of Missouri, Columbia)  
 Agron. J. 61: 777-779. September-October 1969

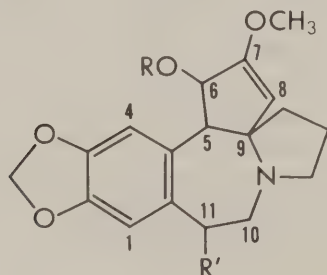
Pericarp thickness of 19 corn (*Zea mays* L.) samples was determined by two methods: microscope and micrometer. Either kernels were frozen and 40  $\mu$  sections cut, stained, and measured with an ocular micrometer or kernels were presoaked and pericarp strips were excised for measurement. The excised strips were measured with a plunger micrometer. The coefficient of rank correlation was +0.85\*\*, indicating good agreement between the two

methods. The microscopic method produced greater thickness values than the micrometer. The lower values were probably due to a certain amount of compression by the micrometer plunger. Germinal-abgerminal ratios differed even though right and left sides of the kernels were similar. The excision method is the more economical of the two and does not need especially trained laboratory technicians.

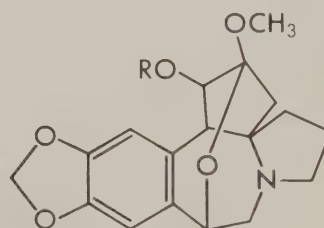
- 2599 • **Structure of Cephalotaxine and Related Alkaloids**  
 R. G. POWELL, D. WEISLEDER, C. R. SMITH, JR., and I. A. WOLFF  
 Tetrahedron Lett. (46): 4081-4084. October 1969

Structures are reported for cephalotaxine (Ia) and three related alkaloids (II, IIIa, and IVa) isolated from the seed of *Cephalotaxus harringtonia* variety *drupacea*. An ester of cephalotaxine (II, C<sub>28</sub>H<sub>37</sub>NO<sub>9</sub>), for which the name harringtonine is proposed, shows significant inhibitory activity against the experimental lymphoid leukemia systems L1210 and P388 in mice at 1.0 mg./kg. Molecular weights of the alkaloids were determined by mass spectrometry, and structural correlations are based primarily on spec-

tral data and on a detailed nuclear magnetic resonance study of the alkaloids and their corresponding acetates (Ib, IIIb, and IVb). An X-ray crystallographic study of cephalotaxine methiodide (D. J. Abraham, R. D. Rosenstein, and E. L. McGandy, University of Pittsburgh) confirmed that Ia is the correct structure for cephalotaxine. Upon basic hydrolysis, II yields Ia and an unidentified acid. The biological activity may be associated either with that acid or the intact molecule II.



- Ia R=H, R'=H  
 Ib R=COCH<sub>3</sub>, R'=H  
 II R=C<sub>10</sub>H<sub>17</sub>O<sub>5</sub>, R'=H



- IIIa R=H, R'=OH  
 IIIb R=COCH<sub>3</sub>, R'=OCOCH<sub>3</sub>  
 IVa R=H  
 IVb R=COCH<sub>3</sub>



2600

- **Aerobic and Facultative Microflora of Fresh and Spoiled Refrigerated Dough Products**

C. W. HESSELTINE, R. R. GRAVES, RUTH ROGERS, and  
H. R. BURMEISTER

Appl. Microbiol. 18(5): 848-853. November 1969

The microbial flora of fresh, unsterile, dough products held at refrigeration temperatures was compared to the microbial flora of the same products that had spoiled spontaneously. Various methods based on selective media were used to determine molds, yeasts, and bacteria present. Except for two special cases in which a yeast and *Penicillium roqueforti* induced spoilage, all the samples deteriorated because of bacterial growth. A total of 1,132 bacterial isolates was subjected to further classification. In the spoiled products 92% of the isolates belonged to the Lactobacillaceae. More than one-half of these (53%) belonged to the genus *Lactobacillus* and an additional 36% were in the genus *Leuconostoc*. In the genus *Leuconostoc* almost all

the strains (94%) were *L. mesenteroides*. The third most common genus present was *Streptococcus*, represented by 3% of the total isolates. A preliminary taxonomic study of the microflora of refrigerated dough products revealed none of the isolates to be indicators of fecal contamination and none to be forms known to produce toxins.

The highest counts encountered in the moist fresh products were up to 200 million lactic acid bacteria per gram in buttermilk biscuits, with a psychrophilic count as high as 4.8 million. In the spoiled samples, the highest total counts were 820 million in buttermilk biscuits. Mold counts were no higher than 1,800, except in the sample ruined by *P. roqueforti* where the count was 130,000 mold colonies.

2601

- **Ring-Opening Reactions of *trans*-Carbonates and Thionocarbonates**

W. M. DOANE, B. S. SHASHA, E. I. STOUT, C. R. RUSSELL, and  
C. E. RIST

Carbohyd. Res. 11(3): 321-329. November 1969

The reaction of methyl 4,6-*O*-benzylidene- $\alpha$ -D-glucopyranoside 2,3-carbonate (I) and of 2,3-thionocarbonate (II) with various nucleophiles was investigated. Under proper conditions, I and II reacted with methanol, benzyl alcohol,  $\alpha$ -toluenethiol, ammonia,

piperidine, and glycine to give the corresponding 2-*O*- and 3-*O*-carbonyl and thiocarbonyl adducts, which were obtained in crystalline form. In each reaction product the 2-isomer was preponderant.

2602

- **Particle Size Analysis of Coatings Materials with the Coulter Counter**

L. H. PRINCEN

Appl. Polym. Symp. (10): 159-176. 1969

A Coulter counter serves for particle size analysis of many coatings materials, such as latexes, oil-in-water emulsions, pigments, and extenders. The counter is suitable to determine quickly any size distribution of dispersed particles between 0.2 and 500  $\mu$  diameter in an electrically conducting liquid. The Coulter counter is used mainly for research purposes, but it has also become a tool for quality con-

trol in production of many powders and emulsions. Topics discussed are: principle of electrozone sizing, precautions to be taken in the use of the instrument, calibration and coincidence correction of the data, particle statistics, and the use of the Coulter counter for studying size distributions of particle systems used in coatings.

- 2603** • **Lipoxidase Deactivation to Improve Stability, Odor and Flavor of Full-Fat Soy Flours**  
G. C. MUSTAKAS, W. J. ALBRECHT, J. E. McGHEE, L. T. BLACK,  
G. N. BOOKWALTER, and E. L. GRIFFIN, JR.  
J. Amer. Oil Chem. Soc. 46(11): 623-626. November 1969

Oxidation of soybean lipids catalyzed by lipoxidase was prevented by heat treatment of soybean meats, which were then ground to give a full-fat soy flour free of rancid odor and flavors. Our previous studies showed that lipids in cracked, dehulled soybeans rapidly oxidized after the lipoxidase system was activated by increasing moisture content to 20%. A series of experiments were conducted in which various heat treatments were evaluated for effectiveness of lipoxidase deactivation. Dry heat to 212° F., steaming, or both, deactivated lipoxidase to give

flours that had low values of peroxide, conjugated diene and free fatty acid and that had good flavors after 2 years of storage. Wet heat alone was also an effective treatment, whereas wet heat preceded by dry heat at 180° F. gave poor flavor stability after 2 years. Gas liquid chromatography studies gave evidence that the rapid formation of volatiles in full-fat soy flours was catalyzed by an enzyme system. A 10-member taste panel was able to detect significant flavor and odor differences between oxidized and nonoxidized samples.

- 2604** • **Two New Members of the Mucorales**  
J. J. ELLIS and C. W. HESSELTINE  
Mycologia 61(5): 863-872. September-October 1969

A new genus is based on a *Mucor*-like species that produces conidia, sporangioles, and columellate, multisporous sporangia. Although the new genus belongs in the Mucoraceae, morphologically it shows some relationship to the family Thamnidaceae. A

new species in the genus *Mucor* is reported belonging in the Petropolitanus section; it emits a strong skunk-like odor and is found only in the southern portion of California. The zygospore stage for both new taxa is illustrated and described.

- 2605** • **Comparison of Glutelin Proteins in Normal and High-Lysine Corn Endosperms**  
J. W. PAULIS, CHRISTOPHER JAMES, and J. S. WALL  
J. Agr. Food Chem. 17(6): 1301-1305. November-December 1969

Purification, structure, and amino acid composition of the glutelins in normal and high-lysine corn endosperms were investigated to determine whether differences in these proteins occur in the two varieties. After removal of saline-soluble proteins, endosperm meals were exhaustively extracted with solutions of either 70% ethanol or 70% ethanol containing 0.5% sodium acetate. The latter solution removed more zein than 70% ethanol alone. Portions of meal residues were then extracted with 0.2% sodium hydroxide to yield glutelin, which appeared partially degraded. To obtain undegraded glutelin, another

residue portion was dispersed in dimethyl sulfoxide solution and its starch digested with  $\alpha$ -amylase. After this glutelin had been reduced and alkylated, additional protein was removed by 70% ethanol extraction. Amino acid composition of the native glutelins and starch-gel electrophoretic patterns of the reduced and alkylated proteins were compared. These data indicated that glutelins of the high-lysine and normal hybrids differed only slightly and that major differences noted in some preparations may result from a greater association of normal glutelin with zein or zein-like proteins.

- 2606 • **A Search for New Fiber Crops. XI. Compositional Characteristics of Illinois Kenaf at Several Population Densities and Maturities**  
T. F. CLARK and I. A. WOLFF  
Tappi 52(11): 2111-2116. November 1969

Physical composition, including fiber content and dimensional characteristics, and contents of selected chemical constituents in the physical components were investigated for kenaf at population densities of 17,800 to 149,000 plants per acre and maturities of 90 to 138 days after seeding, and up to 96 days after frost. Proportions of stalk solids increased with maturity and with population density up to 57,500 plants per acre. Average proportions of bark increased from 17 to 26% as the population density ranged from 17,800 to 57,500 plants per acre. Bast and core fibers, unaffected by maturity and population density, averaged about 21 and 28%, respectively.

Alpha-cellulose (24-28%), lignin (7-10%), and pentosan (9-15%) contents in stalk bottoms increased with maturity, while soluble constituents decreased. Apparent protein contents of 20 to 30% in foliage indicate its potential usefulness for nonpulping purposes. These results suggest that kenaf for pulp be grown to maximum maturity. Storage by standing uncut in the field should be considered where conditions permit. Removal of tops and foliage should be at source, and their utility in animal feeds or as soil amendments should be given attention. Control of stalk diameter for maximum efficiency at harvest is possible by selection of planting density.

- 2607\* • **Microbiological Transformation of Terpenes**  
A. CIEGLER  
In "Fermentation Advances," D. Perlman, editor, pp. 689-714.  
New York. 1969

The microbiological transformation of terpenes other than steroids is reviewed. Only a limited number of studies have been carried out in this area. Transformations successfully performed include hydroxylations, oxidations, reductions, and degrada-

tions. There is need for additional investigation both of the microbiological transformation of biologically active terpenoids and of the mechanisms involved in these transformations.

- 2608 • **Determination of Starch Component of Starch Xanthide in Paper**  
B. S. SHASHA  
Tappi 52(12): 2287-2288. December 1969

A method has been devised for the determination of starch component of starch xanthide in paper. The method is based on destruction of the xanthide group by alkali, treatment of the neutralized sample with

$\alpha$ -amylase, and hydrolysis of the filtrate with acid. Starch content is calculated from the amount of glucose present as determined with glucose oxidase.



2609 • **Di-*O*-Bromoethylidene-D-Mannitol Formation**

H. B. SINCLAIR

J. Org. Chem. 34(12): 3845-3848. December 1969

The stereochemistry and structure of 1,2-*O*-bromoethylidene-D-mannitol and its role in the reaction pathway leading to 1,2:5,6-di-*O*-bromoethylidene-D-mannitol were studied. Nuclear magnetic resonance spectroscopy established that monoacetal formation was from the outset an equilibrium-controlled reaction, giving a 65:35 *cis/trans* ratio. Previously reported work pointed out the high proportion of *cis* rings in the diacetal formation, about 67:33 *cis,cis/cis,trans*. When the latest results are coupled with earlier data, the reaction pathway

can be explained. Monoacetal formation is equilibrium controlled, and the subsequent diacetal formation is kinetically controlled and irreversible because of insolubility. Examination of molecular models suggested that the *cis* preference in the second ring closure may be accounted for by a long-range directional effect transmitted through hydrogen bonding. The unreported 3,4,5,6-tetra-*O*-methyl-D-mannitol was prepared as its crystalline 1,2-di-*O*-*p*-tolylsulfonylester.

2610\* • **A Cyclobutanol Intermediate in the Decomposition of Aldehydes Under Electron Impact. A Further Photochemical Analogy**

CATHERINE FENSELAU,<sup>1</sup> JOHN L. YOUNG,<sup>1</sup> SEYMOUR MEYERSON,<sup>2</sup> WILLIAM R. LANDIS,<sup>3</sup> EDWARD SELKE, and LEONARD C. LEITCH<sup>4</sup>

(<sup>1</sup>Johns Hopkins University, Baltimore, Md.; <sup>2</sup>American Oil Company, Whiting, Ind.; <sup>3</sup>National Institutes of Health, Bethesda, Md.;

<sup>4</sup>National Research Council, Ottawa, Ontario, Canada)

J. Amer. Chem. Soc. 91(24): 6847-6848. November 1969

Loss of C<sub>2</sub>H<sub>4</sub> from the molecular ion of aliphatic aldehyde was previously postulated to be derived from the terminal C<sub>2</sub>H<sub>5</sub> group. Our study, which utilized high and low mass spectra of hexanal, heptanal, and nonanal plus their various deuterated species, showed the P-28 fragment to be C<sub>5</sub>H<sub>10</sub>O<sup>+</sup> and the ethylene lost to comprise essentially those methylenes in the alpha and beta positions in the aldehydes. Previous investigations show close parallels between reactions of acyclic carbonyl compounds under electron impact and UV irradiation.

This suggests that the C<sub>2</sub>H<sub>4</sub> elimination reaction may occur via a cyclobutanol intermediate as in the irradiation of aliphatic ketones. The surmise was tested by comparing the mass spectra, including meta stable peaks, of heptanal to the suspected intermediate, 2-propylcyclobutanol. The mass spectra are similar, which is in accord with a common intermediate. Our findings also suggest that hydrogen migration under electron impact occurs in a discrete primary step which further extends parallels between photochemical and electron impact reactions.

2611 • **A Versatile and Compact Chromatographic Chamber**

J. LEHRFELD

J. Chem. Educ. 46(12): 848. December 1969

A chromatography chamber is described that facilitates the development of 2 X 8-inch thin-layer plates up the 2-inch dimension.

- 2612 • **Oxidative Coupling of Starch Xanthate with Chlorine**  
H. E. SMITH, H. C. KATZ, S. H. GORDON, C. R. RUSSELL,  
and C. E. RIST  
Staerke 21(9): 225-228. September 1969

The oxidative coupling of starch xanthates of degree of substitution (D.S.) 0.05 to 0.21 in 1 and 2% aqueous dispersions with chlorine at 25° C. and pH 5 gave 80% to quantitative yields of xanthides based on starch and afforded sulfur balances of 43 to 80%. In general, xanthide yields, oxidant efficiency, and sulfur balances improved as xanthate D.S. increased

from 0.05 to 0.21. Yields increased sharply to the maximum as xanthate concentration was raised from 0.5 to 1%. Conversion of starch xanthates to starch xanthides was achieved equally well by adding either Clorox or chlorine. Based on cost, chlorine should be more economical.

- 2613\* • **Linseed Oil Coatings Continue To Show Promise**  
J. C. COWAN  
Proc. 38th Annual Flax Inst. of the U.S., Minneapolis,  
Minn., November 21-22, 1968, pp. 36-38. 1969

Methods being used at the Northern Laboratory for preparation of oil-coated pigments and their incorporation into emulsion paints are reviewed. These oil-coated pigments keep the pigment interaction at a minimum and give stable emulsion paints and semi-

glossy films. Use of organic phosphates on zinc oxide improves the wet strength of linseed oil films about fourfold. Linseed oil antispalling and emulsion compounds penetrate test specimens of concrete cured with commercial wax- and resin-curing agents.

- 2614 • **Pilot-Plant Selective Hydrogenation of Soybean Oil: Activation and Evaluation of Copper-Containing Catalysts**  
K. J. MOULTON, D. J. MOORE, and R. E. BEAL  
J. Amer. Oil Chem. Soc. 46(12): 662-666. December 1969

In pilot-plant tests, the linolenate content of soybean oil was reduced to less than 1% without increasing the saturates, by hydrogenation to an IV of about 115 with an active copper-chromite catalyst. The linolenate-linoleate selectivity ratio ( $K_{Le}/K_{Lo}$ ) was from 9 to 12. Several commercial copper-chromite catalysts were used in hydrogenation tests. The activities of four of five commercial catalysts tested were improved to various degrees by heating in air at 350° C. (one was inactive both before and

after heating). Examination by differential thermal analysis (DTA) of each catalyst, just as received and then after being heated at 350° C., demonstrated that heating greatly diminished or removed peak areas from the DTA curve. Studies made with one commercial copper-chromium-barium catalyst showed that heating the catalyst was also necessary to gain maximum linolenate-linoleate selectivity in hydrogenating soybean oil.

- 2615 • **New Sources of 9-D-Hydroxy-*cis*-12-Octadecenoic Acid**  
R. G. POWELL, R. KLEIMAN, and C. R. SMITH, JR.  
Lipids 4(6): 450-453. November 1969

9-D-Hydroxy-*cis*-12-octadecenoic acid has been isolated from three seed oils of the family Apocynaceae: *Holarrhena antidysenterica* (73%), *Nerium oleander* (11%), and *Nerium indicum* (8%). The known occurrence of this acid was previously limited to the genus *Strophanthus* (9-15%).

A mixture of unusual tetra-acid glycerides was isolated from *N. oleander* oil by thin-layer chroma-

tography. Pancreatic lipase hydrolysis of the glyceride mixture showed that 9-D-acetoxy-*cis*-12-octadecenoic acid is esterified exclusively at an  $\alpha$ -glycerol position and that normal fatty acids occupy the remaining two glycerol positions. A portion of the hydroxy acid in *N. indicum* oil was also acetylated; however, no acetate was found in either *H. antidysenterica* or *Strophanthus hispidus* oils.

- 2616 • *Teucrium depressum* Seed Oil: A New Source of Fatty Acids With  $\Delta^5$ -Unsaturation  
C. R. SMITH, JR., R. M. FREIDINGER, J. W. HAGEMANN,  
G. F. SPENCER, and I. A. WOLFF  
Lipids 4(6): 462-465. November 1969

The seed oil of *Teucrium depressum* Small yields two unusual trienoid components, all-*cis*-5,9,12-octadecatrienoic acid (6.7%) and *trans*-5,*cis*-9,*cis*-12-octadecatrienoic acid (2.0%). A third unusual component, identified as *cis*-5,*cis*-9-octadecadienoic acid, also occurs in this oil as a trace constituent.

- 2617 • Glyceride Structure of *Erlangea tomentosa* Seed Oil, a New Source of Vernolic Acid  
B. E. PHILLIPS, C. R. SMITH, JR., and J. W. HAGEMANN  
Lipids 4(6): 473-477. November 1969

(+)-Methyl vernolate constitutes 52% of the methyl esters formed by transmethylation of the oil of *Erlangea tomentosa* seed. Thin-layer chromatography fractionated the oil into trivernolin, divernolins, monovernolins, and nonvernolate glycerides. Pancreatic lipolyses of the monovernolins and the divernolins followed by transmethylation of the monoglycerides indicated the presence of 34 vernolate glycerides above the 0.1% level, grouped as follows:  $\alpha$  monovernolins, 15%;  $\beta$  monovernolins, 3%;  $\alpha$ ,  $\alpha'$ -divernolins, 10%;  $\alpha$ ,  $\beta$ -divernolins, 37%; trivernolin, 16%; and nonvernolate glycerides, 19%.

- 2618 • A Cyanogenetic Lipid from *Cordia verbenacea* DC. Seed Oil  
K. L. MIKOLAJCZAK, D. S. SEIGLER, C. R. SMITH, JR.,  
I. A. WOLFF, and R. B. BATES<sup>1</sup>  
(<sup>1</sup>University of Arizona, Tucson)  
Lipids 4(6): 617-619. November 1969

Cyanogenetic nonglycerol diesters, composed of two fatty acid moieties esterified with an unsaturated five-carbon dihydroxynitrile, constitute 35% of *Cordia verbenacea* DC. (Boraginaceae) seed oil.



## CONTRACT AND GRANT RESEARCH PUBLICATIONS

[Report of research work done by an outside agency under contract with the U.S. Department of Agriculture and supervised by the Northern Utilization Research and Development Division.]

- 215-C      •    **Urethane Coatings Based on Aldehyde Oils**  
A. E. RHEINECK and P. R. LAKSHMANAN  
North Dakota State University, Fargo  
J. Amer. Oil Chem. Soc. 46(9): 452-454. September 1969
  
- 216-C      •    **Reaction of Aldehyde Oils with a Resinous Polyol, Styrene-Allyl Alcohol Copolymer**  
A. E. RHEINECK and P. R. LAKSHMANAN  
North Dakota State University, Fargo  
J. Amer. Oil Chem. Soc. 46(9): 455-458. September 1969
  
- 217-C\*     •    **Production of Vinylic Unsaturation by Partial Dehydrobromination of Vicinal Dibromo Fatty Derivatives**  
A. E. RHEINECK and B. SREENIVASAN  
North Dakota State University, Fargo  
J. Amer. Oil Chem. Soc. 46(9): 464-468. September 1969
  
- 218-C      •    **The Use of Starch as a Reinforcing Filler for Rubber**  
H. L. STEPHENS, R. J. MURPHY, and T. F. REED  
Institute of Polymer Science, University of Akron, Akron, Ohio  
Rubber World 161(2): 77-81. November 1969

[Report of research work done by an outside agency under a grant from the U.S. Department of Agriculture and supervised by the Northern Utilization Research and Development Division.]

- 51-G\*      •    **Reactivity of "V" Amylose Towards Hydrogen Chloride**  
R. P. PANZICA, G. U. YUEN, and B. ZASLOW  
Arizona State University, Tempe  
Carbohydr. Res. 10(3): 343-349. July 1969
  
- 52-G\*      •    **Photolysis of Dimethylthiocarbamates as a Route to Deoxy-Sugars**  
R. H. BELL, D. HORTON, and DIANE M. WILLIAMS  
The Ohio State University, Columbus  
Chem. Commun. (6): 323-324. March 1968

- 53-G\* • **Addition Reactions of a 2,3-Unsaturated Sugar**  
ESTER L. ALBANO, D. HORTON, and J. H. LAUTERBACH  
The Ohio State University, Columbus  
Chem. Commun. (7): 357-358. April 1968
  
- 54-G\* • **Linear Polyaldehydes and Polyalkenes Derived from Starch**  
DAVID M. CLODE, DEREK HORTON, MAKRAM H. MESHREKI,  
and HATSUE SHOJI  
The Ohio State University, Columbus  
Chem. Commun. (13): 694-695. July 1969
  
- 55-G\* • **Methyl Terminal-4-deoxy-, Terminal-4-*O*-ethyl-, and  
Terminal-4-*O*-butyl-malto-oligosaccharides: Synthesis,  
and Hydrolysis with *beta*-Amylase**  
R. E. WING and J. N. BeMILLER  
Southern Illinois University, Carbondale  
Carbohyd. Res. 10(3): 371-377. July 1969
  
- 56-G\* • **Preparation of Alkyl  $\alpha$ -D-Glucopyranosides**  
R. E. WING and J. N. BeMILLER  
Southern Illinois University, Carbondale  
Carbohyd. Res. 10(3): 441-448. July 1969
  
- 57-G\* • **Glycosyl Chlorides Through Reaction with Zinc  
Chloride-Thionyl Chloride**  
VICKY D. GROB, T. G. SQUIRES, and J. R. VERCELLOTTI  
University of Tennessee, Knoxville  
Carbohyd. Res. 10(4): 595-597. August 1969
  
- 58-G\* • **Production of Levoglucosan by Pyrolysis of Carbohydrates**  
C. M. LAKSHMANAN, BENJAMIN GAL-OR, and H. E. HOELSCHER  
University of Pittsburgh, Pittsburgh, Pa.  
Ind. Eng. Chem., Prod. Res. Develop. 8(3): 261-267. September 1969
  
- 59-G • **Reaction of Carbohydrates with Vinyl Ethers; A Differential  
Hydrolysis**  
M. L. WOLFROM, S. S. BHATTACHARJEE, and ROSA M. de  
LEDERKREMER  
The Ohio State University, Columbus  
Carbohyd. Res. 11(1): 148-150. September 1969
  
- 60-G • **Starch Acetals: Acid Sensitivity and Preferred Site  
of Reaction**  
M. L. WOLFROM and S. S. BHATTACHARJEE  
The Ohio State University, Columbus  
Staerke 21(5): 116-118. May 1969
  
- 61-G\* • **Chain Conformation in B-Amylose**  
J. BLACKWELL, A. SARKO, and R. H. MARCHESSAULT  
State University College of Forestry, Syracuse, New York  
J. Mol. Biol. 42(2): 379-383. June 1969

- 62-G\* • **Two Forms of the Glucoamylase of *Aspergillus niger***  
D. R. LINEBACK, I. J. RUSSELL, and C. RASMUSSEN  
University of Nebraska, Lincoln  
Arch. Biochem. Biophys. 134(2): 539-553. November 1969

[Report of research work supported with funds provided by the U.S. Department of Agriculture under the Authority of U.S. Public Law 480, 83rd Congress, and sponsored by the Northern Utilization Research and Development Division.]

- 271-F • **Determination of Carbonyl Groups in Starches by Polarographic Techniques Using *o*-Phenylenediamine. IV. Relation Between the Limiting Current of the Total Quinoxalines and the Molecular Size of Amyloses. A Rapid Determination Method of Molecular Weights of Amylose and Amylopectin**  
M. TAKAGI, T. NISHIO, M. MIZUTANI, N. KAMATANI, Y. IMAOKA, and S. ONO.  
University of Osaka Prefecture, Sakai, Japan  
Staerke 21(1): 2-8. January 1969
- 272-F • **Soybean Saponins. Fate of Ingested Soybean Saponins and the Physiological Aspect of Their Hemolytic Activity**  
BENJAMIN GESTETNER, YEHUDITH BIRK, and YEHUDA TENCER  
The Hebrew University, Rehovoth, Israel  
J. Agr. Food Chem. 16(6): 1031-1035. November-December 1968
- 273-F • **Anionic Graft Polymerization of Formaldehyde on Polyhydroxy Polymers**  
SABAR SASSON and ALBERT ZILKHA  
The Hebrew University, Jerusalem, Israel  
Eur. Polym. J. 5: 315-318. 1969
- 274-F • **Alkali Metal Alkoxide Derivatives of Starch and Dextrin as Initiators of Graft Polymerization of Methacrylonitrile**  
MENASHE TAHAN and ALBERT ZILKHA  
The Hebrew University, Jerusalem, Israel  
Eur. Polym. J. 5: 347-359. 1969
- 275-F • **The Application of the Ptitsyn Theory to Viscosity Data for Amylose and Amylose Acetate in Various Solvents**  
W. BANKS and C. T. GREENWOOD  
University of Edinburgh, Edinburgh, Scotland  
*In* "Polymer. The Science and Technology of Polymers and Biopolymers," vol. 10, pp. 257-264. London. 1969



- 276-F • **Breeding and Stability of Diploid *Saccharomyces rouxii***  
HARUHIKO MORI and HIROSHI ONISHI  
Noda Institute for Scientific Research, Noda-shi,  
Chiba-ken, Japan  
J. Ferment. Technol. 47(6): 394-397. June 1969
  
- 277-F • **Characterization of the *Micrococcus lysodeikticus* Type of Peptidoglycan in Walls of Other *Micrococcaceae***  
J. N. CAMPBELL, MELINA LEYH-BOUILLE, and JEAN-MARIE GHUYSEN  
University of Liege, Liege, Belgium  
Biochemistry 8(1): 193-200. January 1969
  
- 278-F • **The Peptidoglycan in Walls of *Butyribacterium rettgeri***  
MICHELINE GUINAND, JEAN-MARIE GHUYSEN, KARL H. SCHLEIFER, and OTTO KANDLER  
University of Liege, Liege, Belgium  
Biochemistry 8(1): 200-207. January 1969
  
- 279-F • **Structure of the meso-Diaminopimelic Acid Containing Peptidoglycans in *Escherichia coli* B and *Bacillus megaterium* KM**  
JEAN van HEIJENOORT, LYDIA ELBAZ, PHILIPPE DEZELEE, JEAN-FRANCOIS PETIT, EVANGHELOS BRICAS, and JEAN-MARIE GHUYSEN  
University of Liege, Liege, Belgium  
Biochemistry 8(1): 207-213. January 1969
  
- 280-F • **An Improved Technique for the Preparation of *Streptomyces* Peptidases and N-Acetylmuramyl-L-alanine Amidase Active on Bacterial Wall Peptidoglycans**  
JEAN-MARIE GHUYSEN, LOUIS DIERICKX, JACQUES COYETTE, MELINA LEYH BOUILLE, MICHELINE GUINAND, and J. N. CAMPBELL  
University of Liege, Liege, Belgium  
Biochemistry 8(1): 213-222. January 1969
  
- 281-F\* • **Polarographic Studies on Quinoxalines for the Determination of Carbonyl Groups in Starches**  
MASANOSUKE TAKAGI and SOZABURO ONO  
University of Osaka Prefecture, Sakai, Japan  
Bull. Univ. Osaka Pref., Ser. B, 21: 77-122. 1969
  
- 282-F • **Anionic Graft Polymerization of 4-Vinyl Pyridine on Starch and Dextrin**  
SABAR SASSON and ALBERT ZILKHA  
The Hebrew University, Jerusalem, Israel  
Eur. Polym. J. 5: 369-378. 1969

- 283-F • **Soybean Saponins. IX. Studies of their Effect on Birds, Mammals and Cold-Blooded Organisms**  
I. ISHAAYA, YEHUDITH BIRK, A. BONDI, and Y. TENCER  
The Hebrew University of Jerusalem, Rehovoth, Israel  
J. Sci. Food Agr. 20(7): 433-436. July 1969
- 284-F • **Codextrinization of Starch and Gum Karaya. Part I. Fractionation and Chemical Examination of the Codextrin.**  
H. C. SRIVASTAVA, P. P. SINGH, and G. B. DAVE  
Ahmedabad Textile Industry's Research Association,  
Ahmedabad, India  
Staerke 21(5): 123-127. May 1969
- 285-F • **Anionic Graft Polymerization of Acrylonitrile on Starch and Dextrin**  
M. TAHAN, B. YOM-TOV, and A. ZILKHA  
The Hebrew University, Jerusalem, Israel  
Eur. Polym. J. 5: 499-514. 1969
- 286-F • **Studies on Flavor Components in Soybean. Part III. Volatile Fatty Acids and Volatile Amines**  
SOICHI ARAI, HIDEKI SUZUKI, MASAO FUJIMAKI, and YOSITO SAKURAI  
The University of Tokyo, Tokyo, Japan  
Agr. Biol. Chem. (Tokyo) 30(9): 863-868. September 1966
- 287-F • **Studies on Starch-Degrading Enzymes. Part XI. The Stability of Crystalline Sweet Potato Beta-Amylase in Aqueous Solution**  
W. BANKS and C. T. GREENWOOD  
The University of Edinburgh, Edinburgh, Scotland  
Staerke 21(7): 177-180. July 1969
- 288-F • **Decomposition of Soybean Oligosaccharides by Intestinal Bacteria. I. General Introduction and Preliminary Screening of Some Strains of *Escherichia coli* for the Ability of Decomposing Raffinose**  
SIN'ITIRO KAWAMURA, TOSIO MIYAKE, and TEIITI NARASAKI  
Kagawa University, Takamatsu, Japan  
Tech. Bull. Fac. Agr., Kagawa Univ. 20(1): 25-32. December 1968
- 289-F • **Decomposition of Soybean Oligosaccharides by Intestinal Bacteria. II. Comparison of Twenty Strains of *Escherichia coli* for Consuming Each Sugar in the Sugar Mixture Extracted from Defatted Soybean Meal**  
SIN'ITIRO KAWAMURA and TADASI KASAI  
Kagawa University, Takamatsu, Japan  
Tech. Bull. Fac. Agr., Kagawa Univ. 20(1): 33-40. December 1968
- 290-F • **Decomposition of Soybean Oligosaccharides by Intestinal Bacteria. III. Comparison of Eighteen Strains of *Escherichia coli* for Consuming Some Mono- and Oligosaccharides**  
SIN'ITIRO KAWAMURA and TADASI KASAI  
Kagawa University, Takamatsu, Japan  
Tech. Bull. Fac. Agr., Kagawa Univ. 20(1): 41-48. December 1968

- 291-F      Synthesis of 1, 2 -O- and 1, 3-O-Ethylene-D<sub>2</sub>-Glucofuranose**  
H. C. SRIVASTAVA, K. V. RAMALINGAM, and A. S. CHAUDHARI  
Ahmedabad Textile Industry's Research Association, Ahmedabad, India  
Tetrahedron Lett. (31): 2643-2646. July 1969
- 292-F      • Studies of Starches of High Amylose-Content. Part X. An**  
**Improved Method for the Fractionation of Maize and Amylomaize**  
**Starches by Complex Formation from Aqueous Dispersion After**  
**Pretreatment with Methyl Sulphoxide**  
G. K. ADKINS and C. T. GREENWOOD  
The University of Edinburgh, Edinburgh, Scotland  
Carbohydr. Res. 11(2): 217-224. October 1969



July-December 1969

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## PATENTS

[These patents are assigned to the Secretary of Agriculture. Copies of patents may be purchased (50 cents each) from the Commissioner of Patents, U.S. Patent Office, Washington, D.C. 20231. Order by number, do not send stamps.]

### Process for Delayed Production of Japanese Beetle Larvae

GRANT ST. JULIAN, JR.

U. S. Patent 3,463,122. August 26, 1969

Storage of fertile Japanese beetle eggs for as long as 3 weeks to provide an out-of-season supply of newly hatched larvae is made practical by storing the freshly collected eggs at 3-5° C. in 0.85% NaCl

solution for the desired period and then incubating the washed eggs at about 26° C. to induce the hatching of the surviving viable eggs.

### 1,6-Diamino-2,3:4,5-Di-*O*-Benzilidene-1,6-Dideoxygalactitol

WILLIAM A. BLACK, ERIC T. DEWAR, and DAVID RUTHERFORD

U. S. Patent 3,463,790. August 26, 1969

1,6-Diamino-2,3:4,5-di-*O*-benzilidene-1,6-dideoxygalactitol is reacted with sebacoyl dichloride to produce the corresponding nylon-type polyamide containing a carbohydrate residue. Also disclosed are other polyamides which contain carbohydrate

residues. These polyamides have the advantages of not darkening substantially during polymerization and do not develop brittleness that prevent cold drawing of fibers. Some of the polyamides are optically active, rendering them useful as light filters.

### Polarographic Electrode Structure

BERNARD A. WEINER

U. S. Patent 3,476,670. November 4, 1969

A polarographic cathode structure comprising a hypodermic needle having a shaft and an apertured bevel, a longitudinal slit in the wall of the shaft just beyond the bevel, a platinum wire cathode disposed in the shaft, an oxygen-permeable collodion mem-

brane covering the segment of the wire underlying the slit, and a resin seal sealing the aperture at the bevel, whereby only the slit provides access of sample to the cathode.

### Hydrazides of Fatty Quaternary Ammonium Salts of Glycine as Retention Agents

CHARLES L. MEHLTRETTER

U. S. Patent 3,477,904. November 11, 1969

The invention comprises (1) a cationic composition of gelatinized starch and a quaternary ammonium hydrazide salt that contains one fatty aliphatic radical having from 8 to 18 carbon atoms, the gelatinized starch and hydrazide salt being linked through complex formation, and (2) a process for using said composition to increase the retention of starch and

clay in cellulosic fibers in papermaking by adding said composition to cellulosic pulp fibers in water to form an aqueous dispersion, adjusting the pH to about 4 with hydrochloric acid, stirring the resulting mixture, and filtering the mixture to remove the water and recover the fibers and retained starch.

### Levoglucosan Production by Pyrolysis of Pretreated Starches

IVAN A. WOLFF and DAVID W. OLDS

U. S. Patent 3,478,012. November 11, 1969

Yields of levoglucosan from the pyrolysis of dried waxy corn starch, arrowroot starch, or tapioca starch are greatly increased where the waxy starch first has been treated with an SO<sub>2</sub> steep and where the arrowroot or tapioca starches have been steeped

in a dilute solution of a calcium salt such as calcium chloride or calcium acetate. The treatments are beneficial only when applied to the specific starch species, and ordinary pearl corn starch is unaffected.

### Starch Resin Reinforced Rubbers

RUSSELL A. BUCHANAN and CHARLES R. RUSSELL

U. S. Patent 3,480,572. November 25, 1969

High strength white type rubber is produced by milling and vulcanizing the coprecipitate from a preferably synthetic rubber latex that has been fortified by the prior addition per se of certain resin components and either gelatinized starch or starch xanthate having a D. S. of about 0.08.

The instant invention is an extension of the invention taught and claimed in commonly assigned U. S. Patent 3,442,832 of Buchanan *et al.* wherein it is

disclosed that vulcanized white sidewall grade rubber having astonishingly improved mechanical strengths despite the absence of reinforcing carbon blacks or lignin, is obtained by conventionally milling and vulcanizing the crumbs of coprecipitate from a rubber latex that has been augmented prior to coagulation of the latex by the situ addition of critical proportions of either gelatinized starch or of lowly substituted sodium starch xanthate.

### Thermally Stable Stationary Phases for Temperature Programmed Gas Chromatography

VERLE L. DAVISON, EVERETT H. PRYDE, and DANNY J. MOORE

U. S. Patent 3,485,028. December 23, 1969

Improved temperature-programmed gas chromatographic resolutions of mixtures composed of different classes of constituents are obtained even with only one column where the diatomaceous support of

the column has been calcined without flux and then has been coated with certain polyester acetals or polyamide acetals that have then been thermally crosslinked in situ and interbonded thereto.

### Continuous Process for Producing *Xanthomonas* Heteropolysaccharide

SEYMOUR PETER ROGOVIN

U. S. Patent 3,485,719. December 23, 1969

Prolonged steady-state productions of polysaccharide by *Xanthomonas campestris* in a single fermentor using a 2.25 percent corn sugar medium are obtained by fermenting in a batchwise manner

for about 20 hours and then continuously adding sufficient fresh medium to provide an average residence time of 48 to 49 hours to the correspondingly withdrawn effluents.

### Diisocyanate Modified Vegetable Oil Hydroxyl-Terminated Polyesteramide Coatings

LYLE E. GAST and WILMA J. SCHNEIDER

U.S. Patent 3,485,779. December 23, 1969

Baked urethane modified polyesteramide coatings produced by reacting diisocyanate with the hydroxyl-terminated polyesteramide resulting from refluxing particular dicarboxylic compounds with a slight ex-

cess of *N,N*-bis(2-hydroxyethyl) soybean amide or corresponding linseed amide, exhibit greatly improved resistance to dilute alkali.

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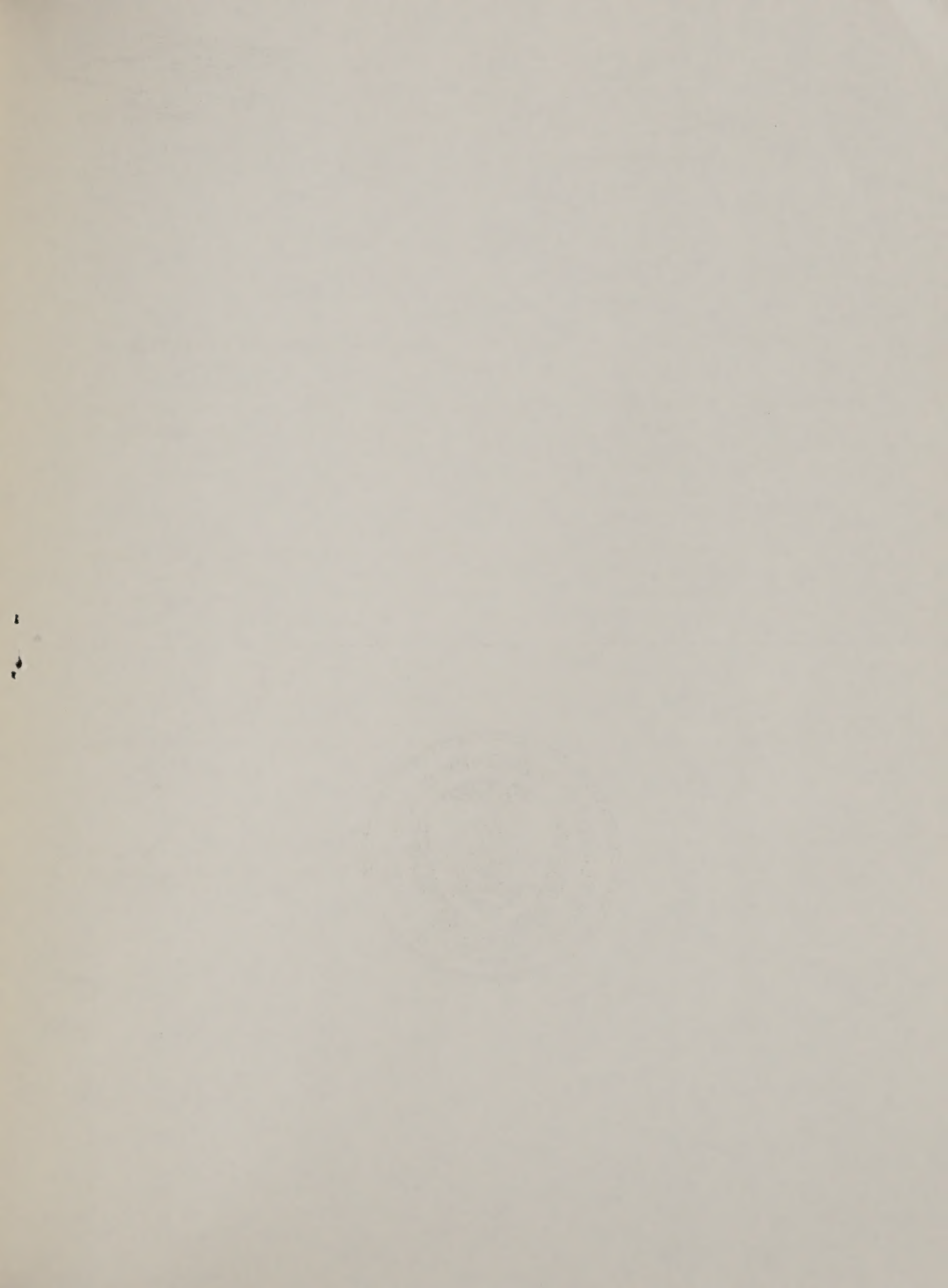
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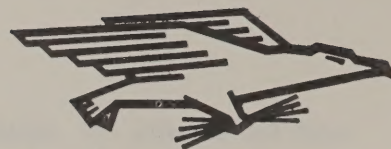
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